federal register

TUESDAY, OCTOBER 16, 1973
WASHINGTON, D.C.

Volume 38 ■ Number 199

PART II

ENVIRONMENTAL PROTECTION AGENCY

■

WATER PROGRAMS

Guidelines Establishing Test Procedures for Analysis of Pollutants

Title 40—Protection of Environment

CHAPTER I—ENVIRONMENTAL PROTECTION AGENCY

SUBCHAPTER D—WATER PROGRAMS

PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

Notice was pubished in the FEDERAL REGISTER issue of June 29, 1973 (38 FR 17318) at 40 CFR 130, that the Environmental Protection Agency (EPA) was giving consideration to the testing procedures required pursuant to section 304(g) of the Federal Water Pollution Control Act Amendments of 1972 (86 Stat. 816, et seq., Pub. L. 92–500 (1972)) hereinafter referred to as the Act. These considerations were given in the form of proposed guidelines establishing test procedures.

Section 304(g) of the Act requires that the Administrator shall promulgate guidelines establishing test procedures for the analysis of pollutants that shall include factors which must be provided in: 1, any certification pursuant to section 401 of the Act, or 2, any permit application pursuant to section 402 of the Act. Such test procedures are to be used by permit applicants to demonstrate that effluent discharges meet applicable pollutant discharge limitations, and by the States and other enforcement activities in routine or random monitoring of effluents to verify effectiveness of pollution control measures.

These guidelines require that discharge measurements, including but not limited to the pollutants and parameters listed in Table I, be performed by the test procedures indicated; or under certain circumstances by other test procedures for analysis that may be more advantageous to use, when such other test procedures have the approval of the Regional Administrator of the Region where such discharge will occur, and when the Director of an approved State National Pollutant Discharge Elimination System (NPDES) Program (hereinafter referred to as the Director) for the State in which such discharge will occur has no objection to such approval.

The list of test procedures in Table I is published herein as final rulemaking and represents major departures from the list of proposed test procedures which was published in 38 FR 17318, dated June 29, 1973. These revisions were made after carefully considering all written comments which were received pertaining to the proposed test procedures. All written comments are on file and available for public review with the Quality Assurance Division, Office of Research and Development, EPA, Washington, D.C.

The principal revisions to the proposed test procedures are as follows:

1. Where several reliable test procedures for analysis are available from the given references for a given pollutant or parameter, each such test procedure has been approved for use for making the measurements required by sections 401 and 402 and related sections of the Act. Approved test procedures have been selected to assure an acceptable level of intercomparability of pollutants discharge data. For several pollutants and parameters it has still been necessary to approve only a single test procedure to assure this level of acceptability. This is a major departure from the proposed test procedures which would have required the use of a single reference method for each pollutant or parameter.

2. Under certain circumstances a test procedure not shown on the approved list may be considered by an applicant to be more advantageous to use. Under guidelines in §§ 136.4 and 136.5 it may be approved by the Regional Administrator of the Region where the discharge will occur, providing the Director has no objections. Inasmuch as there is no longer a single approved reference method against which a comparison can be made, the procedures for establishing such comparisons that were required by the proposed test procedures in § 130.4(b) have been deleted from this final guideline for test procedures for the analysis of pollutants.

3. A mechanism is also provided to assure national uniformity of such approvals of alternate test procedures for the analysis of pollutants. This is achieved through a centralized, internal review within the EPA of all applications for the use of alternate testing procedures. These will be reviewed and approved or disapproved on the basis of submitted information and other available information and laboratory tests which may be required by the Regional Administrator.

As deemed necessary, the Administrator will expand or revise these guidelines to provide the most responsive and appropriate list of test procedures to meet the requirements of sections 304(g), 401 and 402 of the Act, as amended.

These final guidelines establishing test procedures for the analysis of pollutants supersede the interim list of test procedures published in the FEDERAL REGISTER on April 19, 1973 (38 FR 9740) at 40 CFR Part 126 and subsequent procedures published on July 24, 1973 (38 FR 19894) at 40 CFR Part 124. Those regulations established interim test procedures for the submittal of applications under section 402 of the Act. Because of the importance of these guidelines for test procedures for the analysis of pollutants to the National Pollution Discharge Elimination System (NPDES), the Administrator finds good cause to declare that these guidelines shall be effective October 16, 1973.

JOHN QUARLES,
Acting Administrator.

OCTOBER 3, 1973.

PART 136—TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

Sec.

136.1 Applicability.
136.2 Definitions.
136.3 Identification of test procedures.
136.4 Application for alternate test procedures.
136:5 Approval of alternate test procedures.

AUTHORITY: Sec. 304(g) of Federal Water Pollution Control Act Amendments of 1972 86 Stat. 816, et seq., Pub. L. 92–500).

§ 136.1 Applicability.

The procedures prescribed herein shall, except as noted in § 136.5, be used to perform the measurements indicated whenever the waste constituent specified is required to be measured for:

(a) An application submitted to the Administrator, or to a State having an approved NPDES program, for a permit under section 402 of the Federal Water Pollution Control Act as amended (FWPCA), and,

(b) Reports required to be submitted by dischargers under the NPDES established by Parts 124 and 125 of this chapter, and,

(c) Certifications issued by States pursuant to section 401 of the FWPCA, as amended.

§ 136.2 Definitions.

As used in this part, the term:

(a) "Act" means the Federal Water Pollution Control Act, as amended, 33 U.S.C. 1314, et seq.

(b) "Administrator" means the Administrator of the U.S. Environmental Protection Agency.

(c) "Regional Administrator" means one of the EPA Regional Administrators.

(d) "Director" means the Director of the State Agency authorized to carry out an approved National Pollutant Discharge Elimination System Program under section 402 of the Act.

(e) "National Pollutant Discharge Elimination System (NPDES)" means the national system for the issuance of permits under section 402 of the Act and includes any State or interstate program which has been approved by the Administrator, in whole or in part, pursuant to section 402 of the Act.

(f) "Standard Methods" means *Standard Methods for the Examination of Water and Waste Water*, 13th Edition, 1971. This publication is available from the American Public Health Association, 1015 18th St. NW., Washington, D.C. 20036.

(g) "ASTM" means *Annual Book of Standards, Part 23, Water, Atmospheric Analysis, 1972*. This publication is available from the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pennsylvania 19103.

(h) "EPA Methods" means *Methods for Chemical Analysis of Water and Wastes*, 1971, Environmental Protection Agency, Analytical Quality Control Laboratory, Cincinnati, Ohio. This publication is available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402 (Stock Number 5501–0067).

§ 136.3 Identification of test procedures.

Every parameter or pollutant for which an effluent limitation is now specified pursuant to sections 401 and 402 of the Act is named together with test descriptions and references in Table I. The discharge parameter values for which reports are required must be de-

termined by one of the standard analytical methods cited and described in Table I, or under certain circumstances by other methods that may be more advantageous to use when such other methods have been previously approved by the Regional Administrator of the Region in which the discharge will occur, and providing that the Director of the State in which such discharge will occur does not object to the use of such alternate test procedures.

Under certain circumstances the Regional Administrator or the Director in the Region or State where the discharge will occur may determine for a particular discharge that additional parameters or pollutants must be reported. Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator or Director upon the recommendation of the Director of the Methods Development and Quality Assurance Research Laboratory.

TABLE I—LIST OF APPROVED TEST PROCEDURES

Parameter and units	Method	References: Standard methods	References: ASTM	References: EPA methods
General analytical methods:				
1. Alkalinity as $CaCO_3$ mg $CaCO_3$/liter.	Titration: electrometric, manual or automated method—methyl orange.	p. 370	p. 143	p. 6. p. 8.
2. B.O.D. five day mg/liter.	Modified winkler or probe method	p. 489		
3. Chemical oxygen demand (C.O.D.) mg/liter.	Dichromate reflux	p. 495	p. 219	p. 17.
4. Total solids mg/liter	Gravimetric 103–105° C	p. 535		p. 280.
5. Total dissolved (filterable) solids mg/liter.	Glass fiber filtration 180° C			p. 275.
6. Total suspended (nonfilterable) solids mg/liter.	Glass fiber filtration 103–105° C	p. 537		p. 278.
7. Total volatile solids mg/liter.	Gravimetric 550° C	p. 536		p. 282.
8. Ammonia (as N) mg/liter.	Distillation—nesslerization or titration automated phenolate.			p. 134. p. 141.
9. Kjeldahl nitrogen (as N) mg/liter.	Digestion + distillation—nesslerization or titration utomated digestion phenolate.	p. 469		p. 149. p. 157.
10. Nitrate (as N) mg/liter.	Cadmium reduction; brucine sulfate; automated cadmium or hydrazine reduction.	p. 458 p. 461	p. 124	p. 170. p. 175. p. 185.
11. Total phosphorus (as P) mg/liter.	Persulfate digestion and single reagent (ascorbic acid), or manual digestion, and automated single reagent or stannous chloride.	p. 526 p. 532	p. 42	p. 235. p. 246. p. 259.
12. Acidity mg $CaCO_3$/liter	Electrometric end point or phenolphthalein end point.		p. 148	
13. Total organic carbon (TOC) mg/liter.	Combustion—infrared method [1]	p. 257	p. 702	p. 221.
14. Hardness—total mg $CaCO_3$/liter.	EDTA titration; automated colorimetric atomic absorption.	p. 179	p. 170	p. 76. p. 78.
15. Nitrite (as N) mg/liter.	Manual or automated colorimetric diazotization.			p. 185. p. 195.
Analytical methods for trace metals:				
16. Aluminum—total [2] mg/liter.	Atomic absorption	p. 210		p. 98.
17. Antimony—total [2] mg/liter.	Atomic absorption [4]			
18. Arsenic—total mg/liter.	Digestion plus silver diethyldithiocarbamate; atomic absorption.[3]	p. 65 p. 62		p. 13.
19. Barium—total [2] mg/liter.	Atomic absorption [4]	p. 210		
20. Beryllium—total [2] mg/liter.	Aluminon; atomic absorption	p. 67 p. 210		
21. Boron—total mg/liter.	Curcumin	p. 69		
22. Cadmium—total [2] mg/liter.	Atomic absorption; colorimetric	p. 210 p. 422	p. 692	p. 101.
23. Calcium—total [2] mg/liter.	EDTA titration; atomic absorption	p. 84	p. 692	p. 102.
24. Chromium VI mg/liter.	Extraction and atomic absorption; colorimetric.	p. 429		p. 94.
25. Chromium—total [2] mg/liter.	Atomic absorption; colorimetric	p. 210 p. 426	p. 692 p. 403	p. 104.
26. Cobalt—total [2] mg/liter.	Atomic absorption [4]		p. 692	
27. Copper—total [2] mg/liter.	Atomic absorption; colorimetric	p. 210 p. 430	p. 692 p. 410	p. 106.
28. Iron—total [2] mg/liter	do	p. 210 p. 433	p. 692 p. 152	p. 108.
29. Lead—total [2] mg/liter	do	p. 210 p. 436	p. 692	p. 110.
30. Magnesium—total [2] mg/liter.	Atomic absorption; Gravimetric	p. 210 p. 416 p. 201	p. 692	p. 112.
31. Manganese—total [2] mg/liter.	Atomic absorption	p. 210	p. 692	p. 114.
32. Mercury—total mg/liter.	Flameless atomic absorption [5]			
33. Molybdenum—total [2] mg/liter.	Atomic absorption [4]			
34. Nickel—total [2] mg/liter.	Atomic absorption; colorimetric [4]	p. 443	p. 692	
35. Potassium—total [2] mg/liter.	Atomic absorption; colorimetric; flame photometric.	p. 283 p. 285	p. 326	p. 115.
36. Selenium—total mg/liter.	Atomic absorption [3]			
37. Silver—total [2]	Atomic absorption [4]	p. 210		
38. Sodium—total [2] mg/liter.	Flame photometric; atomic absorption	p. 317	p. 326	p. 118.
39. Thallium—total [2] mg/liter.	Atomic absorption [4]			
40. Tin—total [2] mg/liter	do			
41. Titanium—total mg/liter.	do			
42. Vanadium—total [2] mg/liter.	Atomic Absorption; [4] Colorimetric	p. 157		
43. Zinc—total [2] mg/liter	Atomic Absorption; Colorimetric	p. 210 p. 444	p. 692	p. 120.
Analytical methods for nutrients, anions, and organics:				
44. Organic nitrogen (as N) mg/liter.	Kjeldahl nitrogen minus ammonia nitrogen.	p. 468		p. 149.
45. Ortho-phosphate (as P) mg/liter.	Direct single reagent; automated single reagent or stannous chloride.	p. 532	p. 42	p. 235. p. 246. p. 259.
46. Sulfate (as SO_4) mg/liter.	Gravimetric; turbidimetric; automated colorimetric—barium chloranilate.	p. 331 p. 334	p. 51 p. 52	p. 286. p. 288.
47. Sulfide (as S) mg/liter.	Titrimetric—iodine	p. 551		p. 294.
48. Sulfite (as SO_3) mg/liter.	Titrimetric; iodide-iodate	p. 337	p. 261	
49. Bromide mg/liter	do		p. 216	
50. Chloride mg/liter	Silver nitrate; mercuric nitrate; automated colorimetric-ferricyanide.	p. 96 p. 97	p. 23 p. 21	p. 29. p. 31.
51. Cyanide—total mg/liter.	Distillation—silver nitrate titration or pyridine pyrazolone colorimetric.	p. 397	p. 556	p. 41.
52. Fluoride mg/liter	Distillation—SPADNS	p. 171 p. 174	p. 191	p. 64.
53. Chlorine—total residual mg/liter.	Colorimetric; amperometric titration	p. 382	p. 223	
54. Oil and grease mg/liter.	Liquid-Liquid extraction with trichlorotrifluoroethane.	p. 254		
55. Phenols mg/liter	Colorimetric, 4 AAP	p. 502	p. 445	p. 232.
56. Surfactants mg/liter	Methylene blue colorimetric	p. 339	p. 619	p. 131.
57. Algicides mg/liter	Gas chromatography [6]			
58. Benzidine mg/liter	Diazotization—colorimetric [7]			
59. Chlorinated organic compounds (except pesticides) mg/liter.	Gas chromatography [8]			
60. Pesticides mg/liter	Gas chromatography [8]			
Analytical methods for physical and biological parameters:				
61. Color platinum-cobalt units or dominant wave-length, hue, luminance, purity.	Colorimetric; spectrophotometric	p. 160 p. 392		p. 38.
62. Specific conductance mho/cm at 25° C.	Wheatstone bridge	p. 323	p. 163	p. 284.
63. Turbidity jackson units.	Turbidimeter	p. 350	p. 467	p. 308.

See Note at end of Table I

Parameter and units	Method	References		
		Standard methods	ASTM	EPA methods
64. Fecal streptococci bacteria number/100 ml.	MPN; membrane filter; plate count	p. 689 p. 690 p. 691		
65. Coliform bacteria (fecal) number/100 ml.	MPN: Membrane filter	p. 669 p. 684		
66. Coliform bacteria (total) number/100 ml.	do	p. 664 p. 679		
Radiological parameters:				
67. Alpha—total pCi/liter.	Proportional counter; scintillation counter	p. 598	p. 509	
68. Alpha—counting error pCi/liter.	do	p. 598	p. 512	
69. Beta—total pCi/liter.	Proportional counter†	p. 598	p. 478	
70. Beta—counting error pCi/liter.	do	p. 598	p. 478	
71. Radium—total pCi/liter.	Proportional counter; scintillation counter	p. 611 p. 617	p. 674	

[1] A number of such systems manufactured by various companies are considered to be comparable in their performance. In addition, another technique, based on Combustion-Methane Detection, is also acceptable.

[2] For the determination of total metals the sample is not filtered before processing. Choose a volume of sample appropriate for the expected level of metals. If much suspended material is present, as little as 50–100 ml of well-mixed sample will most probably be sufficient. (The sample volume required may also vary proportionally with the number of metals to be determined.)

Transfer a representative aliquot of the well-mixed sample to a Griffin beaker and add 3 ml of concentrated distilled HNO_3. Place the beaker on a hotplate and evaporate to dryness making certain that the sample does not boil. Cool the beaker and add another 3 ml portion of distilled concentrated HNO_3. Cover the beaker with a watch glass and return to the hotplate. Increase the temperature of the hotplate so that a gentle reflux action occurs. Continue heating, adding additional acid as necessary until the digestion is complete, generally indicated by a light colored residue. Add (1:1 with distilled water) distilled concentrated HCl in an amount sufficient to dissolve the residue upon warming. Wash down the beaker walls and the watch glass with distilled water and filter the sample to remove silicates and other insoluble material that could clog the atomizer. Adjust the volume to some predetermined value based on the expected metal concentrations. The sample is now ready for analysis. Concentrations so determined shall be reported as "total".

[3] See D. C. Manning, "Technical Notes", Atomic Absorption Newsletter, Vol. 10, No. 6 p. 123, 1971. Available from Perkin-Elmer Corporation, Main Avenue, Norwalk, Connecticut 06852.

[4] Atomic absorption method available from Methods Development and Quality Assurance Research Laboratory, National Environmental Research Center, USEPA, Cincinnati, Ohio 45268.

[5] For updated method, see: Journal of the American Water Works Association 64, No. 1, pp. 20–25 (Jan. 1972) or ASTM Method D 3223–73, American Society for Testing and Materials Headquarters, 1916 Race St., Philadelphia, Pa. 19103.

[6] Interim procedures for algicides, chlorinated organic compounds, and pesticides can be obtained from the Methods Development and Quality Assurance Research Laboratory, National Environmental Research Center, USEPA, Cincinnati, Ohio 45268.

[7] Benzidine may be estimated by the method of M.A. El-Dib, "Colorimetric Determination of Aniline Derivatives in Natural Waters", El-Dib, M.A., Journal of the Association of Official Analytical Chemists, Vol. 54, No. 6, Nov., 1971, pp. 1383–1387.

†As a prescreening measurement.

§ 136.4 Application for alternate test procedures.

(a) Any person may apply to the Regional Administrator in the Region where the discharge occurs for approval of an alternative test procedure.

(b) When the discharge for which an alternative test procedure is proposed occurs within a State having a permit program approved pursuant to section 402 of the Act, the applicant shall submit his application to the Regional Administrator through the Director of the State agency having responsibility for issuance of NPDES permits within such State.

(c) Unless and until printed application forms are made available, an application for an alternate test procedure may be made by letter in triplicate. Any application for an alternate test procedure under this subchapter shall:

(1) Provide the name and address of the responsible person or firm making the discharge (if not the applicant) and the applicable ID number of the existing or pending permit, issuing agency, and type of permit for which the alternate test procedure is requested, and the discharge serial number.

(2) Identify the pollutant or parameter for which approval of an alternate testing procedure is being requested.

(3) Provide justification for using testing procedures other than those specified in Table I.

(4) Provide a detailed description of the proposed alternate test procedure, together with references to published studies of the applicability of the alternate test procedure to the effluents in question.

§ 136.5 Approval of alternate test procedures.

(a) The Regional Administrator of the region in which the discharge will occur has final responsibility for approval of any alternate test procedure.

(b) Within thirty days of receipt of an application, the Director will forward such application, together with his recommendations, to the Regional Administrator. Where the Director recommends rejection of the application for scientific and technical reasons which he provides, the Regional Administrator shall deny the application, and shall forward a copy of the rejected application and his decision to the Director of the State Permit Program and to the Director of the Methods Development and Quality Assurance Research Laboratory.

(c) Before approving any application for an alternate test procedure, the Regional Administrator shall forward a copy of the application to the Director of the Methods Development and Quality Assurance Laboratory for review and recommendation.

(d) Within ninety days of receipt by the Regional Administrator of an application for an alternate test procedure, the Regional Administrator shall notify the applicant and the appropriate State agency of approval or rejection, or shall specify the additional information which is required to determine whether to approve the proposed test procedure. Prior to the expiration of such ninety day period, a recommendation providing the scientific and other technical basis for acceptance or rejection will be forwarded to the Regional Administrator by the Director of the Methods Development and Quality Assurance Research Laboratory. A copy of all approval and rejection notifications will be forwarded to the Director, Methods Development and Quality Assurance Research Laboratory, for the purposes of national coordination.

[FR Doc.73–21466 Filed 10–15–73;8:45 am]